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Modelling of UV optical ageing of optical fibre fabric coated with TiO_2

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ABSTRACT

Optical fibre fabric developed by Brochier® Technologies Company is studied for water treatment field application. By TiO_2 deposition on this type of textile, the interaction between photocatalyst and UV irradiation can be strongly improved. Indeed the internal irradiation promotes the absorption of light. However, the problem is the ageing of optical fibres induced by UV exposure in the presence of TiO_2 coating.

Two different optical fibre fabrics were compared in term of optical ageing properties. One optical fabric was coated by TiO_2 only; the other fabric was protected by a silica layer before being coated by a photocatalyst. Irradiance measurements of local light emission along ageing process were carried out and were fitted with a theoretical expression of the phenomenon. Macroscopic observations were related to microscopic behaviour on the surface of the fabric. Optical Microscopy (OM) before and after UV exposure confirm the theoretical model suggesting that the ageing induce an increase of existing holes besides appearing of new holes.

These measurements show an important ageing under UV irradiation of the fabrics not protected by a SiO_2 layer, leading to inhomogeneous light distribution at the surface of the textile. In presence of SiO_2 layer between fabrics and TiO_2 layer, no ageing was observed. Moreover, as SiO_2 is transparent to UV, the photocatalyst activity does not change.

1. Introduction

Optical fibres (OF) consist of a core, guiding light, and a cladding. Both parts have a different refractive index in order to allow the propagation of light from one end to the other [1]. Optical fibres are used in many different applications, such as telecommunication, data transfer, illumination and wastewater disinfection [1,2]. Some previous works in our laboratory also showed the possibility to use them to remove pollution by removing optical and mechanical cladding and coating them with TiO_2 [3–7]. Brochier Technologies developed optical fibre weaving solutions for various light applications. Lightex® lighting, protected by several international patents [8,9], consists in the weaving of optical fibres and textile fibres (Polyester textile fibres) with lateral lighting. The optical fibres are in polymethyl methacrylate (PMMA) resin for the core with 480 mm of mean diameter and in fluorinated polymer for the cover with 10 μm of thickness. Thus flexible or rigid illumination surfaces can be created, presenting the advantages to be thin and to require very low power consumption. Side emission of light throughout the fibre is carried out by mechanical micro-texturation, done and patented by Brochier Technologies [8,9]. This technology is nowadays commonly used in application fields as lighting, communication, safety or health. More recently, applications were investigated as pollution control in air environment. So, optical fibre fabrics were coated by a photocatalyst (TiO_2), in order to create an active fabric. In fact, it was previously demonstrated that coating TiO_2 on Brochier Technologies luminous textiles allowed using this type of fabric in environmental field to remove VOC pollutants in air [10]. Effectively, these optical fibre fabrics present some benefits, allowing the development of photocatalytic activated volume against classical activated surface for air treatment, low energy consuming by optimising LED use for photocatalysis and an optimum contact established between light source and photocatalyst support.

However, unlike optical fibres used in our previous works [3–7], those used in luminous textile Brochier Technologies are in PMMA. Because of the organic nature of optical fibres, it was very important

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to study the behaviour of these organic OFs under UVA irradiation exposure in presence of TiO_2 or not. Moreover it is well known that TiO_2 that was coated on textile as a photocatalyst allows to degrade more or less every type of organic compound [11].

PMMA presents the advantage of being inexpensive, of having high ductility, a big core diameter, and shows easy handling characteristics [2,12]. But in spite of these advantages, one of major problem is the photochemical degradation of PMMA optical fibres, especially in presence of UV radiation [2,12–17]. The photodegradation of PMMA under UVC and UVB irradiation have been extensively studied [12–15]. Kaczmarek and Chaberska [15] demonstrated that UVC irradiation at 254 nm induced various surface defects in PMMA film that radically increased surface roughness. Nowadays many studies of photodegradable properties of PMMA were done, concentrating on mechanisms of crosslinking and chain scission of this polymer. Torikai and Hasegawa [14] focused on wavelength sensitivity of photodegradation of PMMA and the effect of the presence of TiO_2-carotene.

Alobaidani et al. [2] demonstrated that the main chain of PMMA can be cleaved by UV irradiation, using a high radiation source in the spectral range from 280 nm to 450 nm. In fact a loss of power of 7% in 13 min was observed. Yu et al. [17] showed the decrease of PMMA film thickness after UV irradiation and the surface modification under UVA irradiation (305–400 nm).

In parallel, photocatalysis belonging to Advanced Oxidative Processes (AOPs) is known to involve the mineralization of a large variety of organic compounds [18,19]. In fact hydroxyl radicals formed at the surface of the photocatalyst, TiO_2, are very reactive and non selective. They are the most oxidant species after fluor radicals. Degradation of polymer by photocatalysis was discussed by Horikoshi et al. [20]. More specifically, the photocatalytic degradation of PMMA was demonstrated by Vinu and Madras [21]. In presence of TiO_2 (Degussa P25) and UVA high pressure mercury vapor lamp (predominant radiation at 365 nm), PMMA and others MMA (methyl methacrylate) co- and homopolymers are degraded randomly along the chain.

All these papers proved the photochemical degradation and photocatalytic degradation of PMMA under UV irradiation. Thus ageing behaviour will be affected by both phenomena.

In this study, we focused on UVA optical ageing of PMMA optical fibre fabrics coating with TiO_2 and its description using mathematical expressions. Especially, evolution of side emission of light was the object of our attention. Moreover ageing will be done in aqueous medium that was not studied before.

2. Materials and methods

2.1. Chemicals

Cristal Activ™ S5-300A TiO_2 suspension was purchased from Millennium Inorganic Chemicals (Thann, France) and was used as received (19.3% wt TiO_2).

Aerodisp® W7622 SiO_2 suspension was purchased from Evonik Degussa GmbH (Rousillon, France) and was used as received (21–23% wt TiO_2, pH = 9.5–10.5). Water used was purified with Milli-Q-Water System.

Formic acid (>99% of purity) was from ACROS Organic. Sulfuric acid (H_2SO_4) for high performance liquid chromatography was purchased from Merck (95–97% of purity).

2.2. Optical fibre fabric and cladding

Optical fibre fabric is produced by Brochier Technologies Company (UVtex®). It is composed of optical fibres in polymethyl methacrylate, made by Mitsubishi (PMMA CK-20 Eska™ fibres) and textile fibres in polyester (Trévira CS™ fibres). These two types of fibres are woven together using Jacquard loom. Warp of fabrics consists only of textile fibres, while weft is composed of both optical and textile fibres. All optical fibres are woven in the same direction, in parallel, and the extremities are gathered, altogether thanks to a cylindrical connector.

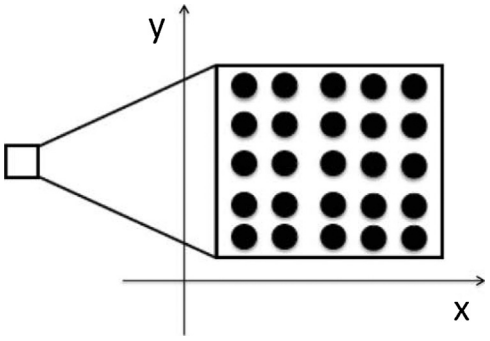

Fig. 1. Measured points used to characterise light emission evolution over exposure time to UV irradiation. 25 measurments dots.

In order to allow light emission over the entire surface of the fabric, a treatment of micro-texturation is done. Micro-texturation is carried out by mechanical abrasion specific technique developed by Brochier Technologies [8,9].

The active surface is a square of 10 cm. In order to have some photocatalytic activity, fabrics are coated with titanium dioxide suspension. Coating is done by soaking optical fabric into TiO_2 suspension, under agitation, during 10 min. Suspension excess is removed by passing fabric though a micro-rolling mill. The same protocol is used for SiO_2 coating.

2.3. Analytical procedures

Fabric surface and coating were analysed by Environmental Scanning Electron Microscopy (ESEM). The microscope was MEB FEI XL30 design by Philips working at low voltage (400 V) in order to know the surface state.

Local emitted light intensities were measured by a CCD Spectrometer. Measurements were performed under radiometric mode, using a CCD Spectrometer Avantes AvaSpec-ULS2048. Before analysis a calibration was carried out by using a Deuterium Halogen Light Source (AvaLight-D(H)-S, Avantes, Apeldoorn, Netherlands). A fibre-optic cable with a cosine corrector (CC-UV/VIS) enables punctual light emission measurements. The cosine corrector diameter is 3.9 mm for a measurement area of 12 mm^2. Spectra in UVA region (320–400 nm) were recorded and processed with AvaSpec 7.0 software via USB. Emitted light intensity was measured through 25 points on the surface of the fabrics (10 cm × 10 cm), as shown on the insert of Fig. 1.

Optical Microscopy Imaging was performed with an open BXFM microscope, having 3 lenses of 10×, 50× and 100× magnification. A camera allowed illuminating fabric in order to visualise and characterise local light emission. A motorize stage enabled to make a planar representation of the surface with an accuracy of 0.1 m. Images acquisition was performed thanks to LabSpec software. Image processing was done through ImageJ software.

2.4. Experimental procedures

Optical ageing of optical fibre fabric was carried out under UV irradiation. Optical fibre fabrics were immersed in purified water. UV irradiation was supported by a LED source directly connected to fabric, emitting at 365 nm with an irradiation of 240 mW/cm^2 (Fig. 2). Optical fibre fabric was subjected to variable exposure time

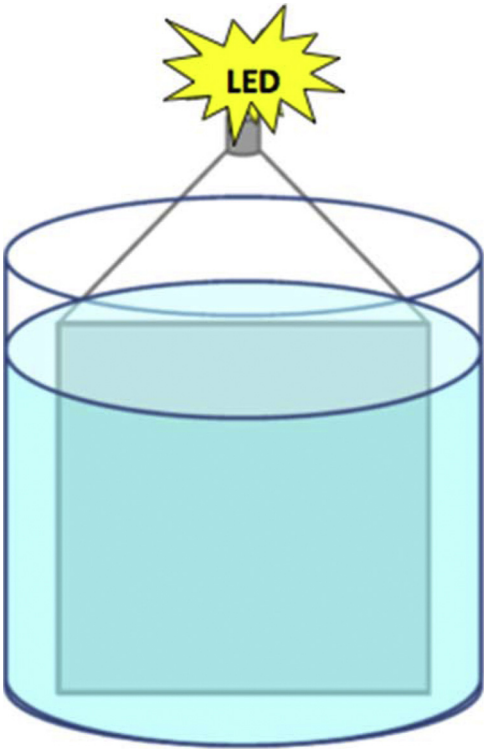

Fig. 2. Optical aging experimental set-up.

in order to follow the emitted light distribution evolution over time exposure to UV.

Radiometric measurements (CCD Spectrometer Avantes AvaSpec-2048) of light emitted over the entire surface were made along exposure time to UV light.

Optical Microscopy pictures were carried out on fabrics before and after exposure to UV ageing in order to measure the hole surface area evolution with time. OM was performed on a 25 mm^2 surface, divided into two zones, one on the top and one on the bottom of the optical fibre.

Photocatalytic degradation tests were done in a cylindrical batch reactor. A formic acid solution of 220 mL at a concentration of 50 ppm was introduced in the reactor and recirculated by an external pump (1650 mL/min). Optical fibre fabric was introduced in the reactor and, after an adsorption phase of 30 min, the UV-LED was switched on and reaction took place during 1.5 h. Samples of reaction medium were removed regularly during the reaction. These samples were analysed by high performance liquid chromatography in order to quantify formic acid degradation over time of reaction.

3. Results and discussion

3.1. Coating characterization and light absorption

In photocatalysis, it is necessary to have interaction between the three main components of the phenomenon: light, photocatalyst and pollutant molecules. Micro-texturated optical fibre fabric allows the light distribution over the overall surface of textile and the activation of TiO_2, coated after previous deposition of SiO_2 or not, on its surface.

As a structural point of view, photocatalytic material coated on textiles is homogeneously spread on surface as shown on Fig. 3a and b (respectively before and after coating). A roughness is visible on optical fibre because of previously made micro-texturated treatment of optical fibre fabric. Optical fibre surface is smoothed

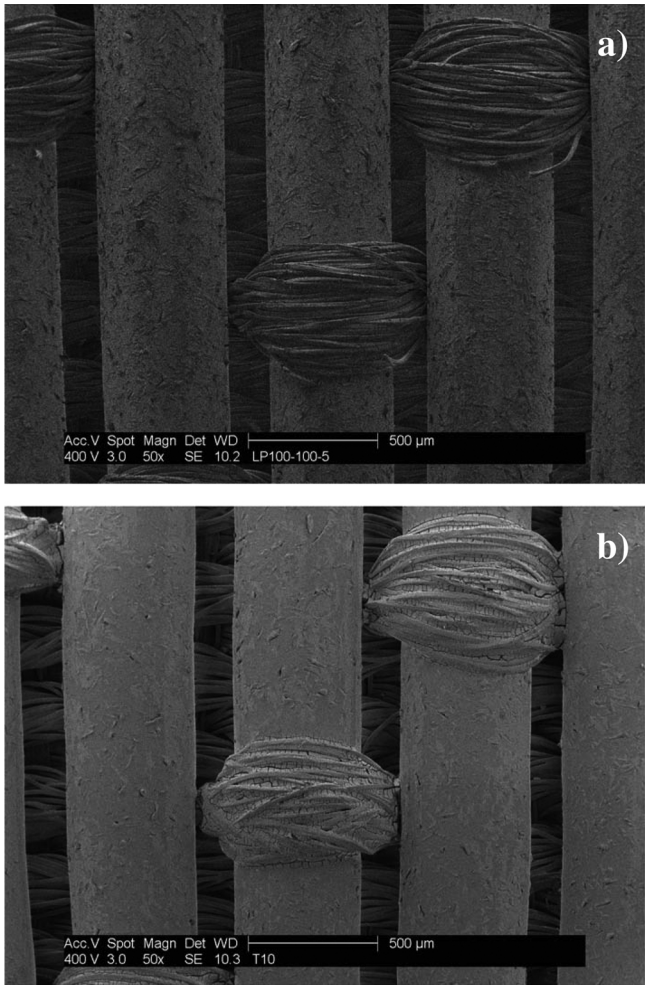

Fig. 3. ESEM picture of optical fibre fabric (a) uncoated, (b) coated by TiO_2.

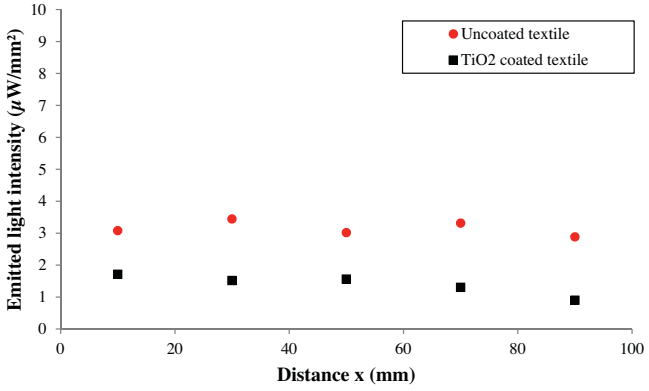

Fig. 4. Emitted light flux of optical fibre fabric. ● uncoated, ■ coated by TiO_2.

after coating, which denotes a photocatalyst coating over the entire surface of fibre. There is an important interaction between light and catalyst leading to a good activation of photocatalyst.

Because TiO_2 is activated by absorbed UV, the amount of absorbed light has to be determined. On Fig. 4, is represented the emitted light on all the surface of the textile uncoated or coated with TiO_2. It has to be noticed that the light was homogeneously distributed on the overall surface before and after coating with TiO_2. This observation is in agreement with a previous work done in our laboratory that demonstrated the homogeneity of light distribution

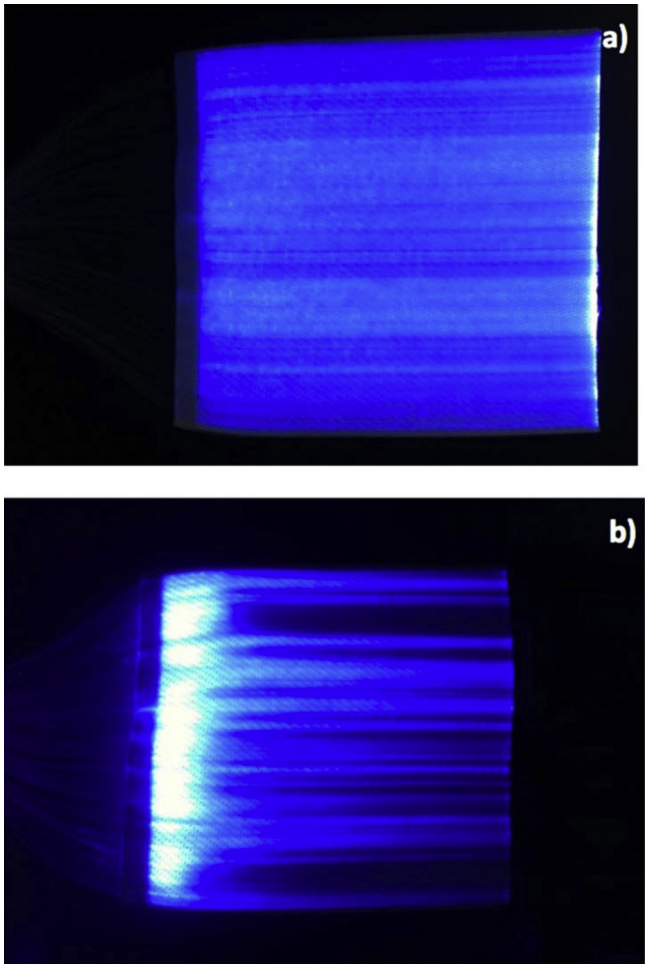

Fig. 5. Photography of light emission distribution on the overall surface of the TiO_2 coated textile (unprotected by silica) before UV irradiation (a), and after 22 days under UV irradiation (b).

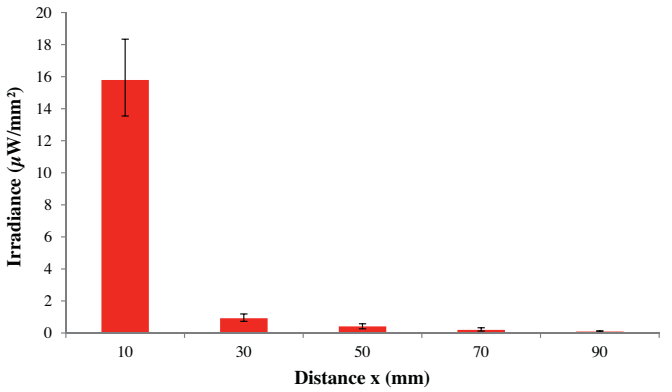

Fig. 6. Irradiance measurements on optical fibre fabric coated by TiO_2 (after 22 days under UV irradiation) over *x*-direction. Bar is the average value of measurements made at distance *x*. The error bar gives maxima and minima values measured at distance *x*.

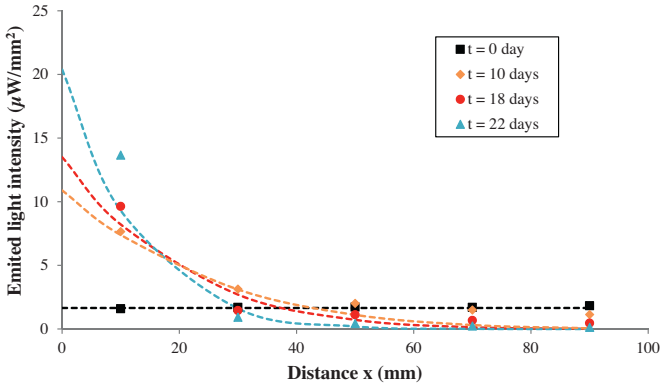

Fig. 7. Emitted light intensity evolution with exposure time to UV, for a fabric coated by TiO_2 (unprotected by SiO_2). ■ at initial time, before UV exposure; ♦ after 10 days under UV irradiation; ● after 18 days under UV irradiation; ▲ after 22 days under UV irradiation.

on the textile surface [10]. The difference observed between the values of irradiance of uncoated and TiO_2 coated textiles is assumed to correspond to the quantity of light absorbed by photocatalyst. About 55% of initial light emission intensity is absorbed by TiO_2. The impact of a SiO_2 layer deposition between fabric and TiO_2 layer was investigated and showed that silica did not modify light absorption, as expected.

3.2. *Impact of UV light on the ageing of uncoated, TiO_2 coated and SiO_2–TiO_2 coated textiles*

3.2.1. *Ageing of TiO_2 coated fabric unprotected by SiO_2 layer*

Fig. 5a and b is photograph of optical fibre fabrics unprotected by SiO_2 under UV irradiation respectively before any irradiation and after 22 days under UV exposition. At initial time, (Fig. 5a) light is relatively homogeneously emitted over the surface. But an important inhomogeneity of the emitted light distribution over the overall surface of fabrics was noted when fabric was exposed to UV irradiation via LED source device, during 22 consecutive days (Fig. 5b). The majority of light is emitted in the first centimetres of the textile.

In order to characterize light emission over textile surface, local emitted light intensities were measured by a CCD Spectrometer. By comparing results before and after 22 days of ageing under UV, it appeared that emitted light intensity was sensibly the same for measurements done at the same distance of the light source (following *y*-axis). So, emitted light intensity is represented (Fig. 6) by taking into account only the *x*-direction evolution that is the direction parallel to optical fibres. Thus the evolution of light flux distribution due to ageing was represented unidirectional.

In order to demonstrate that optical ageing of photocatalytic textile unprotected by SiO_2 layer was due to TiO_2 photocatalytic action and not photochemical autodegradation, an uncoated fabric was exposed to UV irradiation. No modification of emitted light intensity distribution was observed on uncoated fabrics showing clearly that light flux distribution modification is due to the presence of TiO_2.

The evolution of emitted light intensity on the fabric coating by TiO_2, depending on time of exposure to UV, as represented in Fig. 7, illustrates the observation made on photography presented in Fig. 5b for which it can be observed an important light emission from the beginning of fabrics (in the first centimetres). The strong light intensity in first centimetres was brought out by emitted light intensity measurements, giving some very high values, reaching about 3 mW/cm^2 after 25 days of UVA exposition against about 0.1 mW/cm^2 before UVA exposition.

The inhomogeneity of light emission decreases the photocatalytic performance. Actually, the degradation tests performed before and after aging show a decrease of about 20% in the disppearance rate of formic acid (Fig. 8).

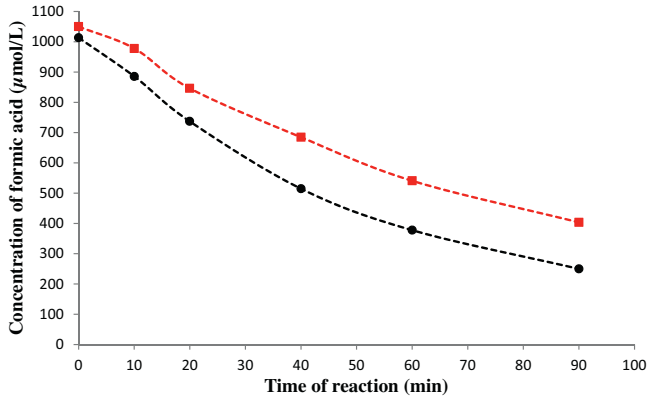

Fig. 8. Evolution of formic acid concentration in function of time of reaction for a luminous textile coated by TiO_2, ● at initial time and ■ after 22 days under UV irradiation.

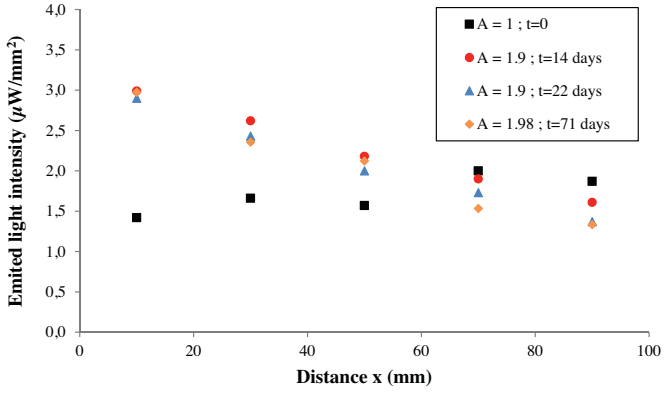

Fig. 9. Emitted light intensity evolution with exposure time to UV, for a fabric coated by SiO_2 and TiO_2 (protected by SiO_2). ■ at initial time, before UV exposure; ♦ after 14 days under UV irradiation; ● after 22 days under UV irradiation; ▲ after 71 days under UV irradiation.

3.2.2. *Impact on the ageing of the deposition of SiO_2 layer before TiO_2 coating*

To avoid the ageing of fabric under UV, leading to an inhomogeneity light leakage on the textile, a silica layer was deposited on fabric before photocatalyst coating. Silica was chosen because of its transparent nature to UV. In fact, silica does not absorb UV, and so does not change the UV absorbance of photocatalyst. In the absence of SiO_2 protecting layer deposition, absorbance values were about 42% (±8%) and with silica coating the absorbance was 47% (±9%). So, taking into account these values, it can be considered that silica did not absorb UV.

Once the fabrics were coated by SiO_2 and TiO_2, they were exposed to UV. Results are presented in Fig. 9. It needs to be noticed that even after a long exposure time, equivalent to 71 days, emitted light profile remained stable and homogeneous over the entire fabric surface. So, the protecting effect of silica against optical ageing in the presence of TiO_2 was demonstrated. This result confirms that drastic ageing of TiO_2 coated fabrics was due to the photocatalytic degradation of PMMA that composed the fibres. By coating textiles with a silica layer, ageing is practically totally avoided.

3.3. *Development of theoretical model for material ageing*

To characterise and describe the ageing behaviour that was observed by irradiance measurements, a theoretical model was developed. The aim is to express the variation of the emitted light intensity at a certain distance from the light source and for a given exposure time to UV. Thus the intensity variation dI, at a time t over a distance dx was expressed by Eq. (1)

$$dI(x,t) = -\alpha N(x)\, s(t)\, I(x)\, dx = -d\varphi(x,t) \tag{1}$$

with $I(x,t)$ the transmitted intensity along the fibre, $\Phi(x,t)$ the emitted intensity of light leaks, α the coefficient of linear light leakage, $N(x)$ the hole density profile at a distance x and $s(t)$ the hole area. By specifying boundary conditions at initial time ($t = 0$) it gave, Eqs. (2)–(4).

$$s(t=0) = s_0 \tag{2}$$

$$\varphi(x, t=0) = \varphi_0 \tag{3}$$

$$dI(x, t=0) = -\varphi_0 dx \Rightarrow I(x, t=0) = I_0 - \varphi_0 x \tag{4}$$

By supposing optical ageing due to photocatalytic degradation did not modify hole density profile during time, and replacing Φ, s and I by their expression in Eq. (1) at $t = 0$, $N(x)$ was given by Eq. (5).

$$N(x) = \frac{\varphi_0}{\alpha s_0 (I_0 - \varphi_0 x)} \tag{5}$$

Then Eq. (1) became for anytime Eq. (6).

$$dI = -\frac{\varphi_0}{s_0 (I_0 - \varphi_0 x)} s(t)\, I(x)\, dx \tag{6}$$

By integrating Eq. (6), the general expression of $I(x)$ is given by:

$$I(x) = I_0 \left(\frac{I_0 - \varphi_0 x}{I_0} \right)^{\frac{s(t)}{s_0}} \tag{7}$$

Actually the emitted flux at instant t, at position x was expressed by (8).

$$\varphi(x,t) = \varphi_0 A \frac{(I_0 - \varphi_0 x)^{A-1}}{{I_0}^{A-1}} \tag{8}$$

in this expression of emitted flux, the parameter $A = s(t)/s_0$ is the ageing parameter and give information about the evolution of the hole surface. The hypothesis made was that holes enlarge with exposure time to UV, involving a more important light leakage that would be concentrated in first centimeters of textiles, as seen before.

3.4. *Experimental measurements fitting with mathematical model*

Once the theoretical model developed, experimental values were fitted with, in order to demonstrate hypothesis made in mathematical model. Emitted light intensity ($\varphi(x)$) at different ageing time (i.e. exposure time to UV) is calculated by using Eq. (8). Parameters φ_0 and I_0 are constants, x is the variable and A, the ageing parameter, is the adjustable parameter, allowing us to express the optical fibre modification occurring under UV irradiation. Fig. 10 shows experimental values fitted with mathematical model. Emitted intensity of light leaks along fibres after 22 days under UV irradiation for TiO_2 coated textile is compared with silica protected material coated by TiO_2. A significant difference in emitted light profile was noted. According to mathematical model, this change is assumed to be due to enlargement of holes that is represented by the change of ageing parameter value (A). In fact, as it can be seen on Fig. 8, the parameter varies from 1 to 12 in the case of unprotected TiO_2 coated textile and varies from 1 to 1.9 in the case of silica protected TiO_2 coated textile. The modification of size of hole, represented by the ageing parameter A, as a function of UV exposure time is given in Fig. 11. A quasi linear increase of hole area can be observed in the absence of SiO_2 layer.

According to these results; it can be said that the mathematical model allows a relatively good expression of the optical ageing of

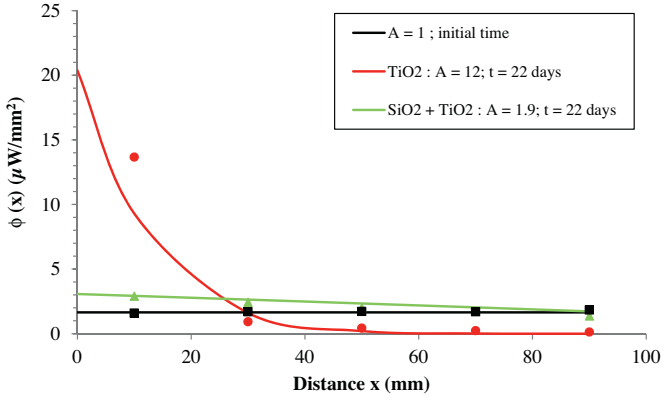

Fig. 10. Theoretical model fitted with experimental values of emitted light intensity along the fibre after exposure to UV during 22 days, for a fabric, ■ uncoated; ● coated by TiO_2; ▲ coated by SiO_2 and TiO_2.

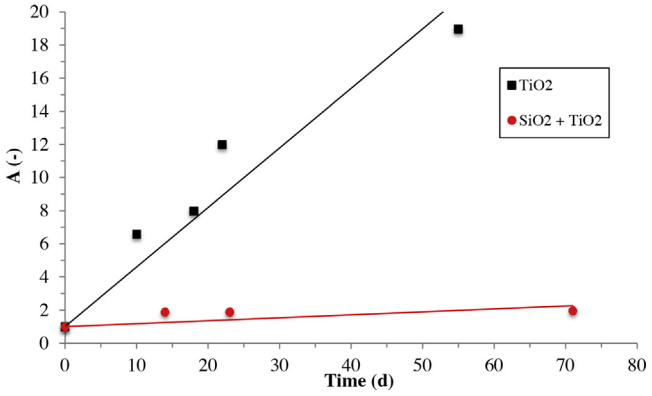

Fig. 11. Hole surface ratio evolution with exposure time to UV, for a fabric, ■ coated by TiO_2; ● coated by SiO_2 and TiO_2.

luminous textile, from a macroscopic point of view. In addition the protecting effect of silica was highlighted.

3.5. Optical microscopy to demonstrate model hypothesis

In order to model light profile evolution over exposure time to UV irradiation, some hypothesis were made, as with ageing time, the hole surface area ratio would increase but hole density profile would be constant with time. These hypotheses seem to correlate the experimental data but microscopic behaviour had to validate macroscopic observations and model hypotheses.

Optical microscopy (OM) was performed to be able to characterise the microscopic behaviour involved by optical ageing phenomenon. This OM observation was done before and after 10 days of UV irradiation in order to compare data and to measure the hole surface area evolution after exposure to UV. Hole density profile would also be determined. Fig. 12a and b, respectively before and after UV exposure (during 10 days) of optical fibre fabric, show an example of microscopic changes on the surface of optical fibres coated by TiO_2. In order to be able to quantify, analyse and interpret the light emission evolution over ageing time, hole surface areas were measured on a same surface size, using ImageJ as picture analysis software.

Results are presented on Fig. 13. A larger number of holes was observed after UV exposure. In fact, an increase of 36% in hole density is obtained.

It is also noted that hole size distribution shows a Gaussian-like profile. So by comparing hole size distribution curves, before and after UV irradiation, it was brought out an enlargement of the

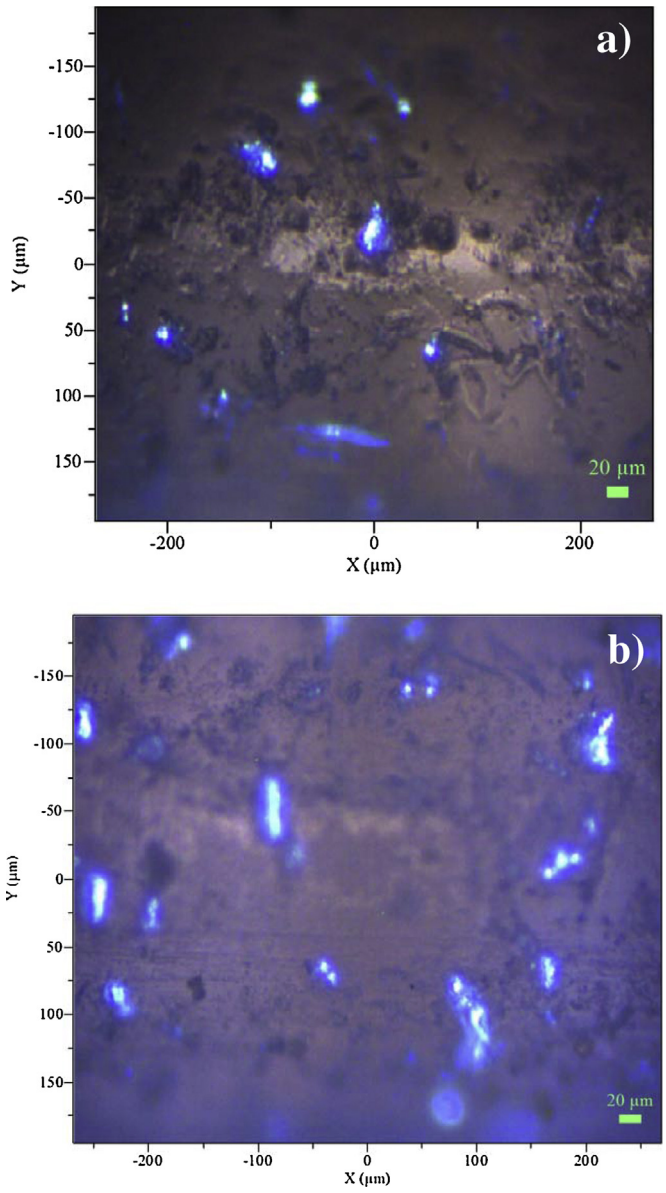

Fig. 12. Optical Microscopy picture before any UV irradiation (a), and after 10 days under UV irradiation (b).

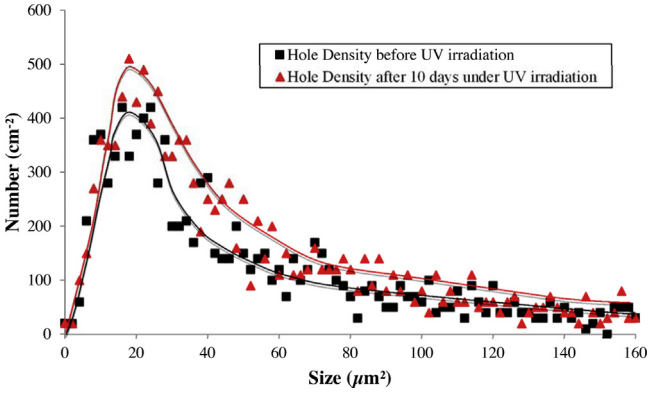

Fig. 13. Evolution of hole density in function of hole surface area between ■ initial state and ▲ after 10 days under UV irradiation.

peak, that reflect an enlargement of hole size. This increase was consistent with the mathematical model developed. But the rise in term of hole density was not taken into account in theoretical model. It also had to be integrated in equation.

It has also to be noted that the evolution of hole surface area with ageing seems to be small because of the relative short irradiation time of UV. Indeed, in order to be able to visualise and measure hole size evolution with ageing time, textile had to be exposed to UV only few days, otherwise it became impossible to do some measure because of a too important light leak in first centimetres of textile and an absence of light on the remaining surface.

4. Conclusions

This work pointed out that:

- There is an optical ageing phenomenon of optical fibre fabrics. Depending on the exposure time to UV, an inhomogeneity of light emission distribution is observed, with majority of light emitted in the first centimetres of the textile.
- The ageing of photocatalytic optical fibre fabric is due to the contact of TiO_2 with organic PMMA optical fibre.
- The presence of a silica layer placed between the textile and the photocatalyst layer highly slows down the luminous textile ageing.

A mathematical model was developed to simulate the optical ageing of optical fibre fabrics. It is based on a constant hole density profile and an enlargement of hole surface area.

The study of hole density and hole size by optical microscopy shows that ageing increases the surface area of holes. However, hole density profile also appears to increase. Nevertheless, the mathematical model is interesting because it can predict the loss and evolution of light emission profile as a function of exposure time to UV light.

Acknowledgements

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